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Description

This invention is concerned with foam control agents and with detergent compositions comprising these foam control agents.

5 Detergent compositions in powder form are used for washing purposes in machines for washing dishes or for laundering of textiles. These compositions generally contain organic surfactants, builders, for example phosphates, bleaching agents and various organic and inorganic additives. The surfactants usually employed in domestic textile washing powders when agitated in an aqueous medium during a washing cycle tend to yield copious quantities of foam. However, presence of excessive amounts of foam
10 during a washing cycle in certain washing machines tends to adversely affect the quality of the wash.

It has become a practice to include in detergent compositions materials which are intended to control the amount of foam produced during a washing cycle. Various materials have been proposed for this purpose, including for example certain silicone antifoams. Silicone antifoams, especially those based on polydimethylsiloxanes, have been found to be particularly useful foam controlling agents in various media.
15 However, generally silicone antifoams, when incorporated in detergent compositions in powder form, appear to lose their effectiveness after prolonged storage in the detergent compositions.

G.B. Patent Specification 1 407 997 is directed to detergent compositions which contain as an essential ingredient a silicone suds controlling agent which is stable on storage. It discloses detergent compositions including a suds controlling component comprising a silicone suds controlling agent and silica or a solid adsorbent releasably enclosed in an organic material which is a water soluble or water dispersible,
20 substantially non-surface active, detergent-impermeable carrier material e.g. gelatin, agar or certain reaction products of tallow alcohol and ethylene oxide. Specification 1 407 997 states that the carrier material contains within its interior substantially all of the silicone suds-controlling agent and effectively isolates it from, i.e. keeps it out of contact with, the detergent component of the compositions. The carrier
25 material is selected such that, upon admixture with water, the carrier matrix dissolves or disperses to release the silicone material incorporated therein to perform its suds-controlling function.

Whilst these materials are satisfactory in many respects it is desirable to enhance the range of materials which may be used to provide storage stable antifoam containing detergent compositions in powder form.

30 G.B. Patent Specification 1 523 957, which relates to detergent compositions containing a silicone foam controlling agent, discloses a powdered or granular detergent composition containing from 0.5 to 20% by weight of a foam control substance which comprises powdered or granular sodium tripolyphosphate, sodium sulphate or sodium perborate having on the surface thereof an organopolysiloxane antifoam agent, which is at least partially enclosed within an organic material which is a mixture of a water insoluble
35 wax having a melting point in the range from above 55°C to below 100°C and a water-insoluble emulsifying agent. We have found that the storage stability of the exemplified detergent compositions disclosed in G.B. Patent Specification 1 523 957 though better than that of detergent compositions where the silicone foam controlling agent is replaced by an organopolysiloxane antifoam agent on its own, is not always satisfactory especially when storage occurs at 40°C over a longer period of time. Other foam control agents
40 have been described in U.S. Patent 3 329 625, E.P. application 76 558, Chemical Abstracts (102) 168703c and U.S. Patent 4 192 761. It is also desirable to reduce the number of those constituents of the foam controlling agent which contribute little or no beneficial effect to the detergent composition when used in a wash cycle.

It is an object of the present invention to provide an improved silicone based foam control agent which
45 is suitable for use in a detergent composition in the form of finely divided powder and is capable of retaining its foam controlling qualities during storage of the detergent composition.

It is also an object of the present invention to provide a foam control agent for inclusion in a detergent composition in powder form, wherein the number of materials used to retain the foam controlling ability of the silicone antifoam is kept to a minimum.

50 We have now found that foam control agents which retain their foam controlling properties during storage in a detergent composition may be wax free and comprise a silicone antifoam and certain organic materials.

The invention provides, in one of its aspects, a particulate foam control agent in finely divided form for inclusion in a detergent composition in powder form, characterised in that the agent is free from
55 monoesters of long chain unbranched fatty acids (C_{24-38}) and alcohols (C_{16-38}) (wax free) and consists of one part by weight of a silicone antifoam comprising a polydiorganosiloxane and a solid silica and from 1 to 5 parts by weight of an organic material having a melting point in the range 50 to 85°C, which is selected from self-emulsifying glyceryl monostearate and one or more monoesters of glycerol and a fatty acid having a carbon chain containing from 12 to 20 carbon atoms.

60 A foam control agent according to the invention is in finely divided form and comprises discrete elements which may be mixed with a detergent composition in powder form in quantities of about 0.1 to 25% by weight of the composition and distributed throughout the composition. During storage at ambient atmosphere conditions at room temperature and at temperatures up to about the melting point of the organic material these discrete elements comprise the organic material and the silicone antifoam. The
65 organic material is selected for its ability to preserve sufficient of the activity of the silicone antifoam

during storage and until required to perform its antifoam function during a washing cycle. Whilst not wishing to be bound by any particular theory, we believe that the organic material acts as a binder or coating to preserve the constitution and disposition of at least a substantial proportion of the discrete elements during storage of the foam control agent in admixture with the detergent composition in powder form. The binder effect of the organic material is unaffected by heating to temperatures lower than its melting point. However, the organic material when heated to a temperature equal to or higher than its melting point becomes liquid and no longer demonstrates the binder effect, thus permitting the previously bound silicone antifoam to be released.

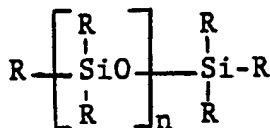
The organic material has a melting point in the range 50 to 85°C. The organic material may comprise a single compound which has a melting point in that range, or a mixture of compounds which has a melting point in that range. Organic materials having a melting point of 50°C or more are chosen in order that the foam control agent may be stable under routine conditions of storage and transportation of a detergent composition in powder form containing it. During summer months, or in warmer countries, during transport or storage the ambient temperature can easily rise to 40°C or more. Also, many housewives store the container of the detergent composition in a room where heat is often produced and temperatures could be in excess of 40°C. Organic materials having a melting point of 85°C or less are selected in order to ensure that any silicone antifoam which remains bound by the organic material when the detergent composition is used in a washing cycle is released at a useful stage in that washing cycle in order to control foaming.

Organic materials suitable for use in a foam control agent according to the invention are selected from one or more monoesters of glycerol and certain fatty acids. Particularly suitable are those organic materials which are at least to some extent water dispersible. The materials selected in the present invention are monoesters of glycerol and aliphatic fatty acids having a carbon chain containing 12 to 20 carbon atoms. Examples of such materials, which are all water insoluble, including glyceryl monolaurate, glyceryl monomyristate, glyceryl monopalmitate and glyceryl monostearate. A more preferred organic material suitable for use in a foam control agent according to the invention is glyceryl monostearate. This material is preferred because of its good performance, easy availability, degree of water dispersibility and suitable melting point. Glyceryl monostearate, having in its pure form a melting point of 82°C (α -ester) or 74°C (β -ester), is commercially available in different grades which are believed to comprise mixtures of the monoester, diester and triester alongside some free glycerol and free stearic acid. Glyceryl monostearate is also available as a non-emulsifying or a self-emulsifying material. The organic material may also be selected from such self-emulsifying glyceryl monostearate. The self-emulsifying glyceryl monostearate comprises also a certain amount of soap. A most preferred organic material for use in a foam control agent according to the invention comprises self-emulsifying glyceryl monostearate. This material is believed to comprise about 30% by weight of the glyceryl monostearate and about 5% by weight of a soap as well as mixtures of diesters and triesters and has a melting point of about 58°C. Self-emulsifying glyceryl monostearate is water dispersible at its melting point of 58°C. Although glyceryl monostearate is surface active it does not appear to interfere with the effectiveness of the silicone antifoam when it is released into the washing liquor.

It is advantageous to provide foam control agents in which not all the silicone antifoam is fully bound, as this appears beneficial to the control of foaming of the detergent composition in the early stage of the wash cycle i.e. before the binding effect of the organic material has been fully disrupted during the washing cycle. In this way one may ensure that sufficient antifoam is available in the early part of the washing cycle for the antifoam to perform its function of controlling the foam level at this stage. This is desirable because excessive foam, even though only present during a part of the wash cycle, usually results in a decrease of the laundering efficiency of the surfactants, due to for example reduced agitation.

The amount of organic material employed in a foam control agent according to the invention is chosen so that when the foam control agent has been added to a detergent composition the composition remains stable upon storage. It is, however, desirable to keep the amount of organic material to a minimum because it is not expected to contribute to the cleaning performance of the detergent composition during a washing cycle. It is also desirable that it is removed from the laundered materials for example with the washing liquor, without causing unacceptable soiling or greying of the laundered materials e.g. through soil redeposition. The amount of organic material employed is best calculated in a weight to weight ratio of organic material to silicone antifoam. A foam control agent according to the invention consists of 1 to 5 parts by weight of the organic material per part by weight of silicone antifoam. Preferably the ratio is from 2:1 to 2.5:1. Ratios below 1:1 may give both manufacturing problems and storage stability problems, while ratios above 5:1 do not seem to contribute any benefit over those obtained at a ratio 5:1 and are commercially less attractive.

A foam control agent according to the invention comprises a silicone antifoam. By the expression silicone antifoam, where used herein, we mean an antifoam compound comprising a polydiorganosiloxane and a solid silica. The polydiorganosiloxane is suitably substantially linear and may have the average formula



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where each R independently can be an alkyl or an aryl radical. Examples of such substituents are methyl, ethyl, propyl, isobutyl and phenyl. Preferred polydiorganosiloxanes are polydimethylsiloxanes having trimethylsilyl endblocking units and having a viscosity at 25°C of from $5 \times 10^{-5} \text{ m}^2/\text{s}$ to $0.1 \text{ m}^2/\text{s}$ i.e. a value of n in the range 40 to 1500. These are preferred because of their ready availability and their relatively low cost. The solid silica of the silicone antifoam can be a fumed silica, a precipitated silica or a silica made by the gelformation technique. The silica particles suitably have an average particle size of from 0.1 to 50 μm , preferably from 1 to 20 μm and a surface area of at least 50 m^2/g . These silica particles can be rendered hydrophobic by treating them with dialkylsilyl groups and/or trialkylsilyl groups bonded directly onto the silica or by means of a silicone resin. We prefer to employ a silica the particles of which have been rendered hydrophobic with dimethyl and/or trimethyl silyl groups. Silicone antifoams employed in a foam control agent according to the invention suitably have an amount of silica in the range of 1 to 30% (more preferably 2 to 15%) by weight of the total weight of the silicone antifoam resulting in silicone antifoams having an average viscosity in the range of from $2 \times 10^{-4} \text{ m}^2/\text{s}$ to $1 \text{ m}^2/\text{s}$. Preferred silicone antifoams may have a viscosity in the range of from $5 \times 10^{-3} \text{ m}^2/\text{s}$ to $0.1 \text{ m}^2/\text{s}$. Particularly suitable are silicone antifoams with a viscosity of $2 \times 10^{-2} \text{ m}^2/\text{s}$ or $4.5 \times 10^{-2} \text{ m}^2/\text{s}$.

Foam control agents according to the invention may be made by any convenient method which enables contacting the silicone antifoam and the organic material in their liquid phase. The conventional procedures for making powders are particularly convenient e.g. spray drying and fluid bed coating procedures. For example the organic material in liquid form and the silicone antifoam in liquid form may be passed into a tower and permitted to form the foam control agent. In one method the silicone antifoam and the organic material are sprayed simultaneously into a spray cooling tower. Upon spraying, small liquid droplets are formed containing the silicone antifoam and the organic material. The droplets cool down as they make their way down the tower. Thus they solidify, forming a particulate finely divided foam control agent according to the invention. The silicone antifoam and the organic material may be mixed prior to spraying, or by contacting the sprayed liquid droplets of both materials, for example by spraying the materials via separate nozzles. The finely divided foam control agent is then collected at the bottom of the tower. Solidification of the droplets may be encouraged, for example by use of a cool air counterstream, thus reducing more quickly the temperature of the droplets. If desired, other ingredients of a detergent composition or component thereof may be passed into the tower e.g. in advance of the organic material and silicone antifoam, so that the foam control agent formed includes carrier particles formed from those ingredients. We prefer to produce the foam control agent by contacting the organic material and the silicone antifoam in their liquid form and passing them onto a fluid bed in which are suspended solid carrier particles. The foam control agent formed includes carrier particles from the fluid bed.

The invention provides in another aspect a particulate foam control agent in finely divided form for inclusion in a detergent composition in powder form, characterised in that the agent is free from monoesters of long chain unbranched fatty acids (C_{24-36}) and alcohols (C_{16-36}) (wax free) and consists of one part by weight of a silicone antifoam comprising a polydiorganosiloxane and a solid silica, from 1 to 5 parts by weight of an organic material having a melting point in the range 50 to 85°C, which is selected from self-emulsifying glyceryl monostearate and one or more monoesters of glycerol and a fatty acid having a carbon chain containing from 12 to 20 carbon atoms, and carrier particles.

These carrier particles may comprise any suitable material but conveniently may be an ingredient or component part of a detergent composition. The carrier particles utilised in the foam control agent provide a solid basis on which the silicone antifoam and the organic material may be deposited and thus provide a dry basis for the silicone antifoam, so that the foam control agent may be a free flowing powder at room temperature and therefore can be easily mixed into a detergent composition in powder form. The carrier particles also bulk up the foam control agent to facilitate the dispersibility of the foam control agent in the powder detergent. Even distribution of the agent in the detergent composition is important since it is desirable that every unit-measure of the detergent composition used by the housewife contains sufficient foam control agent to stop excessive foam formation even though the foam control agent may be employed at levels below 1% by weight of the total detergent composition. Preferably the carrier particles are of water soluble solid powder material which facilitate dispersion of the silicone antifoam in the aqueous liquor during the wash cycle. However, other materials which do not chemically bond with the silicone antifoam may also be used as carrier particles. It is most suitable to choose carrier particles which themselves play an active role in the laundering or washing process. Examples of such materials are zeolites, sodium sulphate, sodium carbonate, carboxymethylcellulose and clay minerals. Such materials are useful as builders, soil suspenders, diluents, softeners etc. in the detergent composition. Preferred carrier particles for use in a foam control agent according to the invention are selected with a view to avoiding settling of the foam control agent to the bottom of a container or detergent composition. Most preferred carrier particles comprise sodium tripolyphosphate (STPP) particles. STPP is preferred because it

has a low bulk density of around 0.5 g/cm³, is water soluble and does not appear to interfere with the effectiveness of the silicone antifoam. A foam control agent according to the invention may comprise carrier particles in an amount of from 60 to 90% by weight based on the total weight of the foam control agent. We prefer to use 70 to 80% of carrier particles by weight of the total foam control agent.

The invention provides in another of its aspects a method of making a particulate wax free foam control agent in finely divided form for inclusion in a detergent composition in powder form characterised in that the silicone antifoam and the selected organic material having a melting point in the range 50 to 85°C are contacted together in their liquid phase and are caused to form a solid in admixture.

In a preferred method according to the invention the silicone antifoam and the organic material are mixed and heated to a temperature above the melting point of the organic material. They may be heated to such temperature before, during or after the mixing stage. The temperature is chosen sufficiently high, for example 90°C, so that the transport from the mixing and/or heating vessel to a spray unit does not cause this temperature to fall below the melting point of the organic material. Any conventional mixing method may be used for the mixing of the silicone antifoam and the organic material for example paddle stirring or ribbon blending. The heated mixture may then be transferred under pressure to a spray nozzle. This can be achieved by any conventional pumping system, but preferably a peristaltic pump is used as this avoids any possible contamination of the mixture with materials from the pump. The pumping rate may vary and can be adapted to the type of spray unit used. The mixture may suitably be pumped at a rate of for example 1.4×10^{-6} m³/s. The spray nozzle and spraying pressure are chosen such that the liquid droplets which are formed are small enough to enable even distribution in a detergent composition. The liquid particles can then fall and deposit themselves in admixture onto a fluid bed of a carrier material, such as the preferred STPP. A foam control agent according to the invention is then collected when the mixture has been sprayed onto the carrier particles.

Foam control agents according to the present invention employ a novel combination of ingredients and enable production of storage stable detergent compositions in powder form without resort to water-soluble or water-dispersible substantially non-surface active, detergent impermeable materials and without the need for adding a water insoluble wax.

Foam control agents according to the invention do not appear to give rise to deposits of the organic material upon textiles laundered with detergent compositions containing these foam control agents in amounts sufficient to control the foam level during laundering operations. An additional advantage of the preferred foam control agents according to the invention is that the amount of organic material introduced into a detergent composition is still acceptable even when a relatively large amount of silicone antifoam is used in the detergent composition.

The present invention also provides a detergent composition in powder form, comprising a detergent component and a foam control agent according to the invention.

A foam control agent according to the invention may be added to the detergent component in a proportion of from 0.1 to 3% by weight based on the total detergent composition weight if no carrier particles are included in the foam control agent. The preferred foam control agents, which include carrier particles, may be added in a proportion of from 0.25 to 25% by weight based on the total detergent composition weight.

Suitable detergent components comprise an active detergent, organic and inorganic builder salts and other additives and diluents. The active detergent may comprise organic detergent surfactants of the anionic, cationic, non-ionic or amphoteric type, or mixtures thereof. Suitable anionic organic detergent surfactants are alkali metal soaps of higher fatty acids, alkyl aryl sulphonates, for example sodium dodecyl benzene sulphonate, long chain (fatty) alcohol sulphates, olefine sulphates and sulphonates, sulphated monoglycerides, sulphated ethers, sulphosuccinates, alkane sulphonates, phosphate esters, alkyl isothionates, sucrose esters and fluorosurfactants. Suitable cationic organic detergent surfactants are alkyl-amine salts, quaternary ammonium salts, sulphonium salts and phosphonium salts. Suitable non-ionic organic surfactants are condensates of ethylene oxide with a long chain (fatty) alcohol or fatty acid, for example C₁₄₋₁₅ alcohol, condensed with 7 moles of ethylene oxide (Dobanol® 45-7), condensates of ethylene oxide with an amine or an amide, condensation products of ethylene and propylene oxides, fatty acid alkylol amides and fatty amine oxides. Suitable amphoteric organic detergent surfactants are imidazoline compounds, alkyl-aminoacid salts and betaines. Examples of inorganic components are phosphates and polyphosphates, silicates, such as sodium silicates, carbonates, sulphates, oxygen releasing compounds, such as sodium perborate and other bleaching agents and zeolites. Examples of organic components are anti-redeposition agents, such as carboxy methyl cellulose (CMC), brighteners, chelating agents, such as ethylene diamine tetraacetic acid (EDTA) and nitrilotriacetic acid (NTA), enzymes and bacteriostats. Materials suitable for the detergent component are well known to the person skilled in the art, and are described in many text books, for example Synthetic Detergents, A. Davidsohn and B. M. Milwidsky, 6th edition, George Godwin (1978).

The following examples are selected to illustrate the invention by way of example. All parts and percentages are expressed by weight unless otherwise stated.

1. Foam control agent production.

All example foam control agents were prepared by stirring 50 g of a silicone antifoam into molten

organic material. The mixture thus formed was heated to 85 or 90°C. This hot liquid mixture was then pumped with a peristaltic pump, via a heat-traced transport line, to the spray head of a fluid bed 'Aeromatic' coating equipment, where it was sprayed at a pressure of 1.2×10^5 Pa through a nozzle of 1.1 mm at a rate of 1.42×10^{-6} m³/s onto a fluid bed of 500 g of STPP (Albright and Wilson, Marchon division). The STPP was kept in the fluid bed by an air pressure at a relative setting of 8 to 10. When all the mixture was sprayed onto the STPP a particulate example foam control agent according to the invention was collected.

Details of the composition (in parts) of each of the example foam control agents are given in Table I.

TABLE I

Example Foam Control Agents

<u>Ingredients</u>	1	2	3	4	5	6	7	8
AF A	5							
AF B		5	5		5	5		
AF C				5			5	5
OM 1	12	12	25	12				
OM 2					12			
OM 3						12		
OM 4							12	
OM 5								12

AF A was a silicone antifoam consisting of a mixture of polydimethylsiloxanes and about 13% by weight of the antifoam of hydrophobic silica. Antifoam A had a viscosity at 25°C of about 2×10^{-2} m²/s.

AF B was a silicone antifoam consisting of a mixture of polydimethylsiloxanes and about 4.5% by weight of the antifoam of hydrophobic silica. Antifoam B had a viscosity at 25°C of about 4.5×10^{-2} m²/s.

AF C was a silicone antifoam consisting of a mixture of polydimethylsiloxanes and about 5% by weight of the antifoam of hydrophobic silica. Antifoam C had a viscosity at 25°C of about 2×10^{-2} m²/s.

OM 1 was self-emulsifying glyceryl monostearate GE 0802 s/e from Croda Chemicals Limited, which is believed to comprise about 30% glyceryl monostearate, a maximum of 7% free glycerine, about 5% soap and a maximum of 2% water. It had a saponification value of about 152 to 160 and a melting point of about 58°C.

OM 2 was glyceryl monostearate GE 0803 (n/e) from Croda Chemicals Limited, which is believed to comprise about 30% glyceryl monostearate, a maximum of 5% free glycerine and a maximum of 2% water. It had a saponification value of about 165 to 175 and a melting point of about 58°C.

OM 3 was glyceryl monostearate GE 3546 (n/e) from Croda Chemicals Limited, which is believed to comprise about 90% glyceryl monostearate, a maximum of 1% free glycerine and a maximum of 2% water. It had a saponification value of about 150 to 165 and a melting point of about 65°C.

OM 4 was glyceryl monomyristate Grindtek® MM 90 from Grindsted Products A/S, which is believed to comprise a minimum of 90% glyceryl monomyristate, a maximum of 4% free glycerol, a maximum of 5% of glyceryl monolaurate and of glyceryl monopalmitate. It had a saponification value of about 180 to 190 and a melting point of about 65°C.

OM 5 was glyceryl monolaurate Grindtek® ML 90 from Grindsted Products A/S, which is believed to comprise a minimum of 90% glyceryl monolaurate, a maximum of 4% free glycerol, a maximum of 5% of glyceryl monocaprate and of glyceryl monomyristate. It had a saponification value of about 200 to 210 and a melting point of about 56°C.

2. Storage stability testing.

A detergent composition was prepared by mixing 9 parts sodium dodecyl benzene sulphonate, 4 parts Dobanol® 45—7, 40 parts sodium tripolyphosphate and 25 parts sodium perborate. This composition is regarded as a basis for a detergent powder composition which may be made up of 100 parts with other ingredients, for example diluents, builders and additives; as these ingredients do not usually tend to contribute significantly to the foam generation of the composition they were not included in the detergent test composition.

The detergent test composition was divided in lots of 78 g to which the foam control agent was then added and mixed in, in proportions sufficient to give, based on the weight of the detergent test composition, the level of silicone antifoam mentioned in Table II or Table III in order to provide sample detergents. Sample detergents 1, 2, 3, 4, 5, 6, 7 and 8 contain respectively first, second, third, fourth, fifth,

sixth, seventh and eighth example foam control agent. Also first and second comparative detergents were prepared consisting of 78 g of the detergent test composition and the silicone antifoam AFA and AFB respectively, as referred to hereinabove in proportions mentioned in Table II.

5 In a first test method a conventional automatic washing machine (Miele® 427) of the front loading type having a transparent door through which clothes may be loaded to the machine was loaded with 3.5 kg of clean cotton fabric. A wash cycle with a prewash and a main wash (95°C) was carried out using one lot of sample detergent for each of the prewash and the main wash, each lot containing 78 g of the detergent test composition. The door of the washing machine was divided in its height by a scale from 0 to 100% with 10% intervals. The level of the top of the foam during the wash cycle was compared with the scale after
10 about 40 minutes of the main wash, when the temperature had reached 90°C, when the rotation drum of the washing machine was stationary and the scale values were recorded.

In a second test method a conventional automatic washing machine (Miele® W 433 de luxe) was used which had a more severe agitation than the machine used in the first test method. This means that a larger amount of foam was generated than in the first method. The test was carried out as in the first method
15 apart from the fact that the prewash was left out.

Sample detergents 1, 2, 3, 5 and 6 and first and second comparative detergents were tested according to the first test method while sample detergents 4, 7 and 8 were tested according to the second test method.

20 One set of sample detergents or comparative detergents was tested immediately after admixture of the foam control agent or of the silicone antifoam to the detergent test composition (initial test), and a second set was stored in closed glass containers at 40°C for 30 days before testing (test after storage). The results are recorded in Tables II and III.

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TABLE II: FIRST TEST METHOD

		% Si-Antifoam Added	FOAM HEIGHT RECORDED (%)	
			Initial Test	Test After Storage
5	<u>Detergents</u>			
	Comparative	0.19	20%	-
10	detergent 1	0.26	NIL	Overflow (9 min) *
15	Sample	0.19	60%	70%
	detergent 1	0.26	30%	70%
		0.32	NIL	30%
20	Comparative	0.19	30%	-
	detergent 2	0.26	NIL	Overflow (7 min) *
25	Sample	0.19	40%	20%
30	detergent 2	0.26	NIL	10%
		0.32	NIL	NIL
35	Sample	0.19	30%	20%
	detergent 3	0.26	NIL	NIL
		0.32	NIL	NIL
40	Sample	0.19	NIL	30%
	detergent 5	0.26	NIL	NIL
45	Sample	0.19	10%	<10%
	detergent 6	0.26	NIL	NIL

* By overflow we mean that the foam came out of the washing machine through a vent at the top. The time at which this occurred, measured from the beginning of the prewash, is given in between brackets.

TABLE III: SECOND TEST METHOD

	<u>Detergents</u>	<u>% Si-Antifoam</u> <u>Added</u>	<u>FOAM HEIGHT RECORDED %</u>	
			<u>Initial</u> <u>Test</u>	<u>Test After</u> <u>Storage</u>
5	Sample	0.19	50%	-
10	detergent 4	0.26	30%	100%
		0.32	50%	50%
		0.38	-	25%
15	Sample	0.26	100%	-
	detergent 7	0.38	25%	100%
20		0.51	30%	75%
	Sample	0.26	100%	-
25	detergent 8	0.38	40%	100%
		0.51	30%	35%

As can be seen from the results shown in Tables II and III the compositions containing a foam control agent according to the invention retain a significant proportion of their foam control ability after prolonged storage.

Claims for the Contracting States: BE DE FR GB IT NL

1. A particulate foam control agent in finely divided form for inclusion in a detergent composition in powder form, characterised in that the agent is free from monoesters of long chain unbranched fatty acids (C_{24-36}) and alcohols (C_{16-36}) (wax free) and consists of one part by weight of a silicone antifoam comprising a polydiorganosiloxane and a solid silica and from 1 to 5 parts by weight of an organic material having a melting point in the range 50 to 85°C, which is selected from self-emulsifying glyceryl monostearate and one or more monoesters of glycerol and a fatty acid having a carbon chain containing from 12 to 20 carbon atoms.

2. A foam control agent according to Claim 1 further characterised in that the organic material is glyceryl monostearate.

3. A foam control agent according to Claim 1 further characterised in that the organic material is self-emulsifying glyceryl monostearate.

4. A foam control agent according to any one of the preceding claims further characterised in that the silicone antifoam comprises a polydimethylsiloxane having trimethylsilyl end-blocking units and a solid silica with a surface area of at least 50 m²/g which has been rendered hydrophobic with dimethyl and/or trimethyl silyl groups.

5. A particulate foam control agent in finely divided form for inclusion in a detergent composition in powder form, characterised in that the agent is free from monoesters of long chain unbranched fatty acids (C_{24-36}) and alcohols (C_{16-36}) (wax free) and consists of one part by weight of a silicone antifoam comprising a polydiorganosiloxane and a solid silica, from 1 to 5 parts by weight of an organic material having a melting point in the range 50 to 85°C, which is selected from self-emulsifying glyceryl monostearate and one or more monoesters of glycerol and a fatty acid having a carbon chain containing from 12 to 20 carbon atoms, and carrier particles.

6. A foam control agent according to Claim 5 further characterised in that the carrier particles comprise sodium tripolyphosphate particles.

7. A method of making a particulate foam control agent according to claim 1 characterised in that one part of a silicone antifoam comprising a polydiorganosiloxane and a solid silica, and from 1 to 5 parts of an organic material having a melting point in the range 50 to 85°C, which is selected from self-emulsifying glyceryl monostearate and one or more monoesters of glycerol and a fatty acid, having a carbon chain containing from 12 to 20 carbon atoms, are contacted together in their liquid phase and are caused to form a solid in admixture in the absence of monoesters of long chain unbranched fatty acids (C_{24-36}) and

alcohols (C₁₈₋₃₆).

8. A method of making a foam control agent according to Claim 5 characterised in that one part of a silicone antifoam comprising a polydiorganosiloxane and a solid silica, and from 1 to 5 parts of an organic material having a melting point in the range 50 to 85°C, which is selected from self-emulsifying glyceryl monostearate and one or more monoesters of glycerol and a fatty acid, having a carbon chain containing from 12 to 20 carbon atoms, are mixed together and are sprayed in the form of liquid droplets onto a fluidised bed of carrier particles, onto which the liquid droplets solidify.

9. A detergent composition in powder form comprising a detergent component and a foam control agent according to any one of Claims 1 to 6.

Claims for the Contracting State: AT

1. A method of making a particulate foam control agent in finely divided form for inclusion in a detergent composition in powder form, which is free from monoesters of long chain unbranched fatty acids (C₂₄₋₃₆) and alcohols (C₁₈₋₃₆) (wax free) and consists of one part by weight of a silicone antifoam comprising a polydiorganosiloxane and a solid silica and from 1 to 5 parts by weight of an organic material having a melting point in the range 50 to 85°C, which is selected from self-emulsifying glyceryl monostearate and one or more monoesters of glycerol and a fatty acid having a carbon chain containing from 12 to 20 carbon atoms characterised in that one part of a silicone antifoam comprising a polydiorganosiloxane and a solid silica and from 1 to 5 parts of an organic material having a melting point in the range 50 to 85°C, which is selected from self-emulsifying glyceryl monostearate and one or more monoesters of glycerol and a fatty acid, having a carbon chain containing from 12 to 20 carbon atoms are contacted together in their liquid phase and are caused to form a solid in admixture in the absence of monoesters of long chain unbranched fatty acids (C₂₄₋₃₆) and alcohols (C₁₈₋₃₆).

2. A method of making a foam control agent according to Claim 1 further characterised in that the silicone antifoam and the organic material are sprayed simultaneously and are caused to solidify by cooling.

3. A method of making a particulate foam control agent in finely divided form for inclusion in a detergent composition in powder form, which is free from monoesters of long chain unbranched fatty acids (C₂₄₋₃₆) and alcohols (C₁₈₋₃₆) (wax free) and consists of one part by weight of a silicone antifoam comprising a polydiorganosiloxane and a solid silica and from 1 to 5 parts by weight of an organic material having a melting point in the range 50 to 85°C, which is selected from self-emulsifying glyceryl monostearate and one or more monoesters of glycerol and a fatty acid having a carbon chain containing from 12 to 20 carbon atoms characterised in that one part of a silicone antifoam comprising a polydiorganosiloxane and a solid silica and from 1 to 5 parts of an organic material having a melting point in the range 50 to 85°C, which is selected from self-emulsifying glyceryl monostearate and one or more monoesters of glycerol and a fatty acid, having a carbon chain containing from 12 to 20 carbon atoms are mixed together and are sprayed in the form of liquid droplets onto a fluidised bed of carrier particles, onto which the liquid droplets solidify.

4. A method of making a foam control agent according to any one of the preceding claims further characterised in that the organic material is glyceryl monostearate.

5. A method of making a foam control agent according to any one of the preceding claims further characterised in that the organic material is self-emulsifying glyceryl monostearate.

6. A method of making a foam control agent according to any one of the preceding claims further characterised in that the silicone antifoam comprises a polydimethylsiloxane having trimethylsilyl end-blocking units and a solid silica with a surface area of at least 50 m²/g which has been rendered hydrophobic with dimethyl and/or trimethyl silyl groups.

7. A method of making a foam control agent according to Claim 3 further characterised in that the carrier particles comprise sodium tripolyphosphate particles.

Patentansprüche für die Vertragsstaaten: BE DE FR GB IT NL

1. Teilchenförmiger Schaumregulator in feinverteilter Form für die Aufnahme in einer Detergens-Zusammensetzung in Pulverform, dadurch gekennzeichnet, daß der Regulator frei von Monoestern langkettiger, unverzweigter Fettsäuren (C₂₄₋₃₆) und Alkoholen (C₁₈₋₃₆) (wachsfrei) ist und aus 1 Gewichtsteil eines Silikon-Antischaummittels, das ein Polydiorganosiloxan und ein festes Siliciumdioxid enthält, und 1 bis 5 Gewichtsteilen eines organischen Materials mit einem Schmelzpunkt im Bereich von 50 bis 85°C besteht, wobei das organische Material von selbstemulgierendem Glycerylmonostearat und einem oder mehreren Monoestern von Glycerin und einer Fettsäure mit einer Kohlenstoffkette, die 12 bis 20 Kohlenstoffatome enthält, ausgewählt ist.

2. Schaumregulator nach Anspruch 1, dadurch gekennzeichnet, daß das organische Material Glycerylmonostearat ist.

3. Schaumregulator nach Anspruch 1, dadurch gekennzeichnet, daß das organische Material ein selbstemulgierendes Glycerylmonostearat ist.

4. Schaumregulator nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß das Silikon-Antischaummittel ein Polydimethylsiloxan mit Trimethylsilyl-Endblockeinheiten und ein festes Siliciumdioxid mit einer Oberfläche von wenigstens 50 m²/g enthält, wobei das Siliciumdioxid mit Dimethyl- und/oder Trimethylsilylgruppen hydrophob gemacht worden ist.

5. Teilchenförmiger Schaumregulator in feinverteilter Form für die Aufnahme in einer Detergens-Zusammensetzung in Pulverform, dadurch gekennzeichnet, daß der Regulator frei von Monoestern langkettiger, unverzweigter Fettsäuren (C₂₄₋₃₆) und Alkoholen (C₁₆₋₃₆) (wachsfrei) ist und aus 1 Gewichtsteil eines Silikon-Antischaummittels, das ein Polydiorganosiloxan und ein festes Siliciumdioxid enthält, 1 bis 5 Gewichtsteilen eines organischen Materials mit einem Schmelzpunkt im Bereich von 50 bis 85°C und Trägerteilchen besteht, wobei das organische Material von selbstemulgierendem Glycerylmonostearat und einem oder mehreren Monoestern von Glycerin und einer Fettsäure mit einer Kohlenstoffkette, die 12 bis 20 Kohlenstoffatome enthält, ausgewählt ist.

6. Schaumregulator nach Anspruch 5, dadurch gekennzeichnet, daß die Trägerteilchen Natriumtripolyphosphatteilchen umfassen.

7. Verfahren zur Herstellung eines teilchenförmigen Schaumregulators nach Anspruch 1, dadurch gekennzeichnet, daß man 1 Teil eines Silikon-Antischaummittels, das ein Polydiorganosiloxan und ein festes Siliciumdioxid enthält, und 1 bis 5 Teile eines organischen Materials mit einem Schmelzpunkt im Bereich von 50 bis 85°C, das von selbstemulgierendem Glycerylmonostearat und einem oder mehreren Monoestern von Glycerin und einer Fettsäure mit einer Kohlenstoffkette, die 12 bis 20 Kohlenstoffatome enthält, ausgewählt ist, miteinander in ihrer flüssigen Phase in Berührung bringt und in Vermischung die Bildung eines Feststoffs bewirkt, wobei Monoester von langkettigen unverzweigten Fettsäuren (C₂₄₋₃₆) und Alkoholen (C₁₆₋₃₆) abwesend sind.

8. Verfahren zur Herstellung eines Schaumregulators nach Anspruch 5, dadurch gekennzeichnet, daß man 1 Teil eines Silikon-Antischaummittels, das ein Polydiorganosiloxan und ein festes Siliciumdioxid enthält, und 1 bis 5 Teile eines organischen Materials mit einem Schmelzpunkt im Bereich von 50 bis 85°C, das von selbstemulgierendem Glycerylmonostearat und einem oder mehreren Monoestern von Glycerin und einer Fettsäure mit einer Kohlenstoffkette, die 12 bis 20 Kohlenstoffatome enthält, ausgewählt ist, zusammen vermischt und in Form von flüssigen Tröpfchen auf ein Fließbett von Trägerteilchen versprüht, auf denen die flüssigen Tröpfchen sich verfestigen.

9. Detergens-Zusammensetzung in Pulverform, die eine Detergenz-Komponente und eine Schaumregulator gemäß einem der Ansprüche 1 bis 6 enthält.

Patentansprüche für den Vertragsstaat: AT

1. Verfahren zur Herstellung eines teilchenförmigen Schaumregulators in feinverteilter Form für die Aufnahme in einer Detergens-Zusammensetzung in Pulverform, wobei der Regulator frei von Monoestern langkettiger, unverzweigter Fettsäuren (C₂₄₋₃₆) und Alkoholen (C₁₆₋₃₆) (wachsfrei) ist und aus 1 Gewichtsteil eines Silikon-Antischaummittels, das ein Polydiorganosiloxan und ein festes Siliciumdioxid enthält, und aus 1 bis 5 Gewichtsteilen eines organischen Materials mit einem Schmelzpunkt im Bereich von 50 bis 85°C besteht, wobei das organische Material von selbstemulgierendem Glycerylmonostearat und einem oder mehreren Monoestern von Glycerin und einer Fettsäure mit einer Kohlenstoffkette, die 12 bis 20 Kohlenstoffatome enthält, ausgewählt ist, dadurch gekennzeichnet, daß man 1 Teil eines Silikon-Antischaummittels, das ein Polydiorganosiloxan und ein festes Siliciumdioxid enthält, und 1 bis 5 Teile eines organischen Materials mit einem Schmelzpunkt im Bereich von 50 bis 85°C, das von selbstemulgierendem Glycerylmonostearat und einem oder mehreren Monoestern von Glycerin und einer Fettsäure mit einer Kohlenstoffkette, die 12 bis 20 Kohlenstoffatome enthält, ausgewählt ist, zusammen in ihrer flüssigen Phase in Berührung bringt und im vermischten Zustand die Bildung eines Feststoffs bewirkt, wobei Monoester von langkettigen unverzweigten Fettsäuren (C₂₄₋₃₆) und Alkoholen (C₁₆₋₃₆) abwesend sind.

2. Verfahren zur Herstellung eines Schaumregulators nach Anspruch 1, dadurch gekennzeichnet, daß man das Silikon-Antischaummittel und das organische Material gleichzeitig versprüht und die Verfestigung durch Kühlung bewirkt.

3. Verfahren zur Herstellung eines teilchenförmigen Schaumregulators in feinverteilter Form für die Aufnahme in einer Detergens-Zusammensetzung in Pulverform, wobei der Regulator frei von Monoestern langkettiger, unverzweigter Fettsäuren (C₂₄₋₃₆) und Alkoholen (C₁₆₋₃₆) (wachsfrei) ist und aus 1 Gewichtsteil eines Silikon-Antischaummittels, das ein Polydiorganosiloxan und ein festes Siliciumdioxid enthält, und 1 bis 5 Gewichtsteilen eines organischen Materials mit einem Schmelzpunkt im Bereich von 50 bis 85°C besteht, wobei das organische Material von selbstemulgierendem Glycerylmonostearat und einem oder mehreren Monoestern von Glycerin und einer Fettsäure mit einer Kohlenstoffkette, die 12 bis 20 Kohlenstoffatome enthält, ausgewählt ist, dadurch gekennzeichnet, daß man 1 Teil eines Silikon-Antischaummittels, das ein Polydiorganosiloxan und ein festes Siliciumdioxid enthält, und 1 bis 5 Teile eines organischen Materials mit einem Schmelzpunkt im Bereich von 50 bis 85°C, das von selbstemulgierendem Glycerylmonostearat und einem oder mehreren Monoestern von Glycerin und einer Fettsäure mit einer Kohlenstoffkette, die 12 bis 20 Kohlenstoffatome enthält, ausgewählt ist, zusammen vermischt und in Form von flüssigen Tröpfchen auf ein Fließbett von Trägerteilchen versprüht, auf denen die flüssigen Tröpfchen sich verfestigen.

4. Verfahren zur Herstellung eines Schaumregulators nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß das organische Material Glycerylmonostearat ist.

5. Verfahren zur Herstellung eines Schaumregulators nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß das organische Material ein selbstemulgierendes Glycerylmonostearat ist.

6. Verfahren zur Herstellung eines Schaumregulators nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß das Silikon-Antischaummittel ein Polydimethylsiloxan mit Trimethylsilyl-Endblockeinheiten und ein festes Siliciumdioxid mit einer Oberfläche von wenigstens 50 m²/g umfaßt, wobei das Siliciumdioxid mit Dimethyl- und/oder Trimethylsilylgruppen hydrophob gemacht worden ist.

7. Verfahren zur Herstellung eines Schaumregulators nach Anspruch 3, dadurch gekennzeichnet, daß die Trägerteilchen Natriumtripolyphosphatteilchen enthalten.

Revendications pour les Etats contractants: BE DE FR GB IT NL

1. Un agent inhibiteur de mousse particulaire sous forme finement divisée à incorporer dans une composition détergente sous forme de poudre, caractérisé en ce que l'agent est exempt de monoesters d'acides gras non ramifiés à longue chaîne (C₂₄—C₃₆) et d'alcools (C₁₆—C₃₆) (sans cires) et se compose d'une partie en poids d'un antimousse siliconé comprenant un polydiorganosiloxane et une silice solide et de 1 à 5 parties en poids d'une substance organique ayant un point de fusion situé dans l'intervalle de 50 à 85°C, qui est choisie parmi un monostéarate de glycérile auto-émulsionnant et un ou plusieurs monoesters du glycérol et d'un acide gras dont la chaîne carbonée compte 12 à 20 atomes de carbone.

2. Un agent inhibiteur de mousse selon la revendication 1, caractérisé de plus en ce que la substance organique est du monostéarate de glycérile.

3. Un agent inhibiteur de mousse selon la revendication 1, caractérisé de plus en ce que la substance organique est du monostéarate de glycérile autoémulsionnant.

4. Un agent inhibiteur de mousse selon l'une quelconque des revendications précédentes, caractérisé de plus en ce que l'antimousse siliconé comprend un polydiméthylsiloxane ayant des motifs triméthylsilyles de blocage d'extrémités et une silice solide ayant une surface spécifique d'au moins 50 m²/g qui a été rendue hydrophobe par des groupes diméthyl- et/ou triméthylsilyles.

5. Un agent inhibiteur de mousse particulaire sous forme finement divisée à incorporer dans une composition détergente sous forme de poudre, caractérisé en ce que l'agent est exempt de monoesters d'acides gras non ramifiés à longue chaîne (C₂₄—C₃₆) et d'alcools (C₁₆—C₃₆) (sans cires) et se compose d'une partie en poids d'un antimousse siliconé comprenant un polydiorganosiloxane et une silice solide, de 1 à 5 parties en poids d'une substance organique ayant un point de fusion situé dans l'intervalle de 50 à 85°C, qui est choisie parmi un monostéarate de glycérile auto-émulsionnant et un ou plusieurs monoesters du glycérol et d'un acide gras dont la chaîne carbonée compte 12 à 20 atomes de carbone, et de particules de support.

6. Un agent inhibiteur de mousse selon la revendication 5, caractérisé de plus en ce que les particules de support comprennent des particules de tripolyphosphate de sodium.

7. Un procédé pour fabriquer un agent inhibiteur de mousse particulaire selon la revendication 1, caractérisé en ce qu'une partie d'un antimousse siliconé comprenant un polydiorganosiloxane et une silice solide, et 1 à 5 parties d'une substance organique ayant un point de fusion situé dans l'intervalle de 50 à 85°C, qui est choisie parmi un monostéarate de glycérile auto-émulsionnant et un ou plusieurs monoesters du glycérol et d'un acide gras dont la chaîne carbonée compte 12 à 20 atomes de carbone, sont mis en contact ensemble dans leur phase liquide et sont amenés à former un solide en mélange en l'absence de monoesters d'acides gras non ramifiés à longue chaîne (C₂₄—C₃₆) et d'alcools (C₁₆—C₃₆).

8. Un procédé pour fabriquer un agent inhibiteur de mousse selon la revendication 5, caractérisé en ce qu'une partie d'un anti-mousse siliconé comprenant un polydiorganosiloxane et une silice solide, de 1 à 5 parties d'une substance organique ayant un point de fusion situé dans l'intervalle de 50 à 85°C, qui est choisie parmi un monostéarate de glycérile auto-émulsionnant et un ou plusieurs monoesters du glycérol et d'un acide gras dont la chaîne carbonée compte 12 à 20 atomes de carbone, sont mélangés ensemble et sont atomisés sous forme de gouttelettes liquides sur un lit fluidisé de particules de support sur lequel les gouttelettes liquides se solidifient.

9. Une composition détergente sous forme de poudre, comprenant un constituant détergent et un agent inhibiteur de mousse selon l'une quelconque des revendications 1 à 6.

Revendications pour l'Etat contractant: AT

1. Un procédé pour fabriquer un agent inhibiteur de mousse particulaire sous forme finement divisée à incorporer dans une composition détergente sous forme de poudre, qui est exempt de monoesters d'acides gras non ramifiés à longue chaîne (C₂₄—C₃₆) et d'alcools (C₁₆—C₃₆) (sans cires) et se compose d'une partie en poids d'un antimousse siliconé comprenant un polydiorganosiloxane et une silice solide et de 1 à 5 parties en poids d'une substance organique ayant un point de fusion situé dans l'intervalle de 50 à 85°C, qui est choisie parmi un monostéarate de glycérile auto-émulsionnant et un ou plusieurs monoesters du glycérol et d'un acide gras dont la chaîne carbonée compte 12 à 20 atomes de carbone, caractérisé en ce

qu'une partie d'un antimousse siliconé comprenant un polydiorganosiloxane et une silice solide et 1 à 5 parties d'une substance organique ayant un point de fusion situé dans l'intervalle de 50 à 85°C, qui est choisie parmi un monostéarate de glycéryle auto-émulsionnant et un ou plusieurs monoesters du glycérol et d'un acide gras dont la chaîne carbonée compte 12 à 20 atomes de carbone, sont mis en contact
5 ensemble dans leur phase liquide et sont amenés à former un solide en mélange en l'absence de monoesters d'acides gras non ramifiés à longue chaîne ($C_{24}-C_{36}$) et d'alcools ($C_{16}-C_{36}$).

2. Un procédé pour fabriquer un agent inhibiteur de mousse selon la revendication 1, caractérisé en ce que l'antimousse siliconé et la substance organique sont atomisés simultanément et sont amenés à se solidifier par refroidissement.

10 3. Un procédé de fabriquer un agent inhibiteur de mousse particulaire sous forme finement divisée à incorporer dans une composition détergente sous forme de poudre, qui est exempt de monoesters d'acides gras non ramifiés à longue chaîne ($C_{24}-C_{36}$) et d'alcools ($C_{16}-C_{36}$) (sans cires) et se compose d'une partie en poids d'un antimousse siliconé comprenant un polydiorganosiloxane et une silice solide et de 1 à 5 parties en poids d'une substance organique ayant un point de fusion situé dans l'intervalle de 50 à
15 85°C, qui est choisie parmi un monostéarate de glycéryle auto-émulsionnant et un ou plusieurs monoesters du glycérol et d'un acide gras dont la chaîne carbonée compte 12 à 20 atomes de carbone, caractérisé en ce qu'une partie d'un antimousse siliconé comprenant un polydiorganosiloxane et une silice solide et 1 à 5 parties d'une substance organique ayant un point de fusion situé dans l'intervalle de 50 à 85°C, qui est choisie parmi un monostéarate de glycéryle auto-émulsionnant et un ou plusieurs monoesters du glycérol et d'un acide gras dont la chaîne carbonée compte 12 à 20 atomes de carbone, sont mélangés ensemble et
20 sont atomisés sous la forme de gouttelettes liquides sur un lit fluidisé de particules de support, sur lequel les gouttelettes se solidifient.

4. Un procédé pour fabriquer un agent inhibiteur de mousse selon l'une quelconque des revendications précédentes, caractérisé de plus en ce que la substance organique est le monostéarate de glycéryle.

25 5. Un procédé pour fabriquer un agent inhibiteur de mousse selon l'une quelconque des revendications précédentes, caractérisé de plus en ce que la substance organique est un monostéarate de glycéryle auto-émulsionnant.

6. Un procédé pour fabriquer un agent inhibiteur de mousse selon l'une quelconque des revendications précédentes, caractérisé de plus en ce que l'antimousse siliconé comprend un polydiméthylsiloxane
30 ayant des motifs triméthylsilyles de blocage d'extrémités et une silice solide ayant une surface spécifique d'au moins 50 m²/g, qui a été rendu hydrophobe par des groupes diméthyl- et/ou triméthylsilyles.

7. Un procédé pour fabriquer un agent inhibiteur de mousse selon la revendication 3, caractérisé de plus en ce que les particules de support comprennent des particules de tripolyphosphate de sodium.

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